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(54) Title: ORAL COMPOSITION

(57) Abstract: An oral composition comprising picolinic acid and a peroxy species or equivalent source thereof, characterised in that the molar ratio of picolinic acid to peroxy species or equivalent source thereof is from 1:30 to 100:1.

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ORAL COMPOSITION

The present invention relates to an oral composition according to the preamble of claim 1.

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Peroxyl species, in particular hydrogen peroxide and sources thereof, are well-known antimicrobial agents, although they have not been widely used in deodorant products for use on the human body. It is also known that the stability of such materials can be improved by the addition of small amounts of transition metal chelator. The use of dipicolinic acid for such purpose is described in several publications, for example EP 666,307 A (Procter and Gamble). Dipicolinic acid is also claimed to improve the antimicrobial effect hydrogen peroxide - see EP 845,526 A (Eka Chemicals).

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Picolinic acid has been described in WO9007501 (Solvay Interlox) as a stabiliser for percarboxylic acid bleaching compositions. The picolinic acid is added in minor amounts to sequester transition metals which catalyse peroxygen compound decomposition. Picolinic acid is also listed as an optional component for this purpose in EP 1,074,607 (Ausimont S.p.A.).

20

We have discovered that synergistic mixtures of picolinic acid and a peroxyl species or equivalent source thereof can achieve the target of providing an excellent antimicrobial benefit and subsequent protection against plaque, caries and gingivitis. We have also found that synergistic mixtures of picolinic acid and a peroxyl species or equivalent source

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thereof can achieve the target of providing an excellent tooth whitening benefit.

None of the prior art discloses or suggests the products of  
5 the present invention, nor the excellent antimicrobial and tooth whitening benefits obtainable by the use thereof.

We have found that use of a synergistic mixture of picolinic acid and a peroxy species or equivalent source thereof  
10 gives an excellent tooth whitening antimicrobial benefit- much greater than that obtained from either of the two components individually. In addition, the addition of picolinic acid at such a level as to give said synergistic benefit enables less of the peroxy species or equivalent  
15 source thereof to be used in the product - a significant benefit for products used on the human body.

Thus, in a first aspect of the invention, there is provided an oral composition according to claim 1.

20

In a second aspect of the present invention, there is provided a cosmetic method of whitening the teeth comprising the use of picolinic acid and a peroxy species or equivalent source thereof.

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In a third aspect of the present invention, there is provided a method for the manufacture of an oral composition for the treatment of gingivitis, caries and/or plaque by mixing picolinic acid and a peroxy species or equivalent  
30 source thereof with a cosmetically acceptable carrier material.

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The antimicrobial and tooth whitening benefit derived from use of the present invention may be gained by independent application of picolinic acid and the peroxy species or equivalent source thereof. Such application may be concurrent or consecutive, provided that the treated substrate experiences the presence of both components at the same time. When the components are applied from independent compositions, it is preferred that the product also comprises a means for, and/or instruction for, both of the compositions to be applied to the human body. Where the peracid and picolinic acid are stored independently from one another the formulatory requirements may be such that the stability of either is optimised. For example, if one formulation comprises a peracid such as PAP the pH of the formulation will be between 3.5 and 5, preferably 4. The formulation of the formulation comprising picolinic acid will then be modified such that when the two components are mixed they provide an oral composition according to the invention and having an acceptable pH of between 6.5 and 7.5.

It is preferred that the picolinic acid and the peroxy species or equivalent source thereof are applied from the same composition. A preferred product according to the invention is a single composition comprising both the picolinic acid and the peroxy species or equivalent source thereof.

The cosmetic method of gaining an antimicrobial benefit on the human body referred to in the second aspect of the

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present invention preferably uses the picolinic acid and the peroxy species or equivalent source thereof at a molar ratio of from 1:30 to 100:1. This molar ratio, and that present in the composition according to the first aspect of the invention, is more preferably from 1:20 to 50:1 and most preferably from 1:10 to 20:1.

Picolinic acid is an essential component in the products of the invention. It may be used at a level of from 0.01% to 10%, particularly from 0.1% to 8%, and especially from 0.15% to 5%, by weight based on total weight of the composition. The picolinic acid may be used in its acid form or as its salt. Suitable salts include alkaline metal salts, alkaline earth metal salts, amine salts, and quaternary ammonium salts. When the picolinic acid is partially or totally in its salt form, the preferred amount is equivalent to the aforementioned preferred levels, on a molar basis.

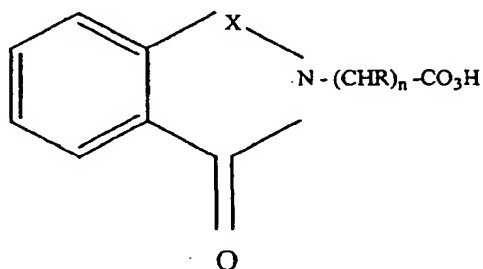
The products of the invention comprise a peroxy species per se or a material that generates a peroxy species in situ. A peroxy species is one that comprises a peroxy (-O-O-) group. Examples of suitable peroxy species include hydrogen peroxide and peracids. Examples of equivalent sources thereof are compounds that produce hydrogen peroxide on dissolution in water, such as sodium perborate monohydrate, sodium perborate tetrahydrate, sodium percarbonate and percarbamide (urea-hydrogen peroxide addition compound). Further examples are enzymatic hydrogen peroxide generating systems such as peroxidases, oxidases and other oxido-reductase enzyme systems, in conjunction

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with their appropriate substrates. Preferred products comprise a peracid, in particular a peracid of formula (1)

(1)

5



in which R is hydrogen or C<sub>1</sub>-C<sub>4</sub> alkyl; n is 1 to 5; and X is C = O or SO<sub>2</sub>.

- 10 The peroxy amido phthalamides of formula 1) are known per se and have been described in EP-A-325,288 and EP-A-325,289. A preferred compound of this formula is N-phthalimido hexanoic peroxy acid ("PAP") of formula 1), in which R = H, n = 5 and X = C=O. An example of a compound according to formula 1)
- 15 wherein x = SO<sub>2</sub> is saccharin-perhexanoic acid ("saccharin PAP"), as described in EP-A-485,927. Alternatively, a compound that produces hydrogen peroxide on dissolution in water, rather than hydrogen peroxide itself. Suitable peracids include any of those mentioned in WO96/05802 the
- 20 contents relating to peracids being hereby incorporated by reference.

Particularly preferred products comprise a compound that produces hydrogen peroxide on dissolution in water,

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especially products from which hydrogen peroxide is absent, prior to application.

The amount of peroxy species or equivalent source thereof in compositions of the invention may range from 0.0001% to 5%, more preferably from 0.001 to 1.5%, most preferably from 0.005% to 0.5%, by weight based on total weight of the composition.

10 Where the oral composition is to be used for its antimicrobial effect the composition may also comprise a transition metal chelator. Whilst picolinic acid is a transition metal chelator, performance may be increased by the use of a further material of this class, in particular a material having a high binding constant for iron (III); that is to say, a binding constant for iron (III) of greater than  $10^{15}$ , preferably greater than  $10^{20}$ , and most preferably greater than  $10^{26}$ . A particularly preferred material of this class is diethylenetriaminepentaacetic acid (DTPA). Salts of such materials may also be employed, suitable salts being analogous to those described as suitable picolinic acid salts (*vide supra*). The total amount of additional transition metal chelator and salt thereof that is employed is typically from 0.1% to 5%, in particular from 0.2% to 3%, and especially from 0.4% to 2% by weight of the composition of which it is a part.

The oral composition according to the invention comprise further ingredients which are common in the art, such as:

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antimicrobial agents, e.g. Triclosan, chlorhexidine, copper-  
, zinc- and stannous salts such as zinc citrate, zinc  
sulphate, zinc glycinate, sodium zinc citrate and stannous  
pyrophosphate, sanguinarine extract, metronidazole,

5 quaternary ammonium compounds, such as cetylpyridinium  
chloride; bis-guanides, such as chlorhexidine digluconate,  
hexetidine, octenidine, alexidine; and halogenated  
bisphenolic compounds, such as 2,2' methylenebis-(4-chloro-  
6-bromophenol);

10

anti-inflammatory agents such as ibuprofen, flurbiprofen,  
aspirin, indomethacin etc.;

anti-caries agents such as sodium- and stannous fluoride,  
15 aminefluorides, sodium monofluorophosphate, sodium trimeta  
phosphate and casein;

plaque buffers such as urea, calcium lactate, calcium  
glycerophosphate and strontium polyacrylates;

20

vitamins such as Vitamins A, C and E;

plant extracts;

25 desensitising agents, e.g. potassium citrate, potassium  
chloride, potassium tartrate, potassium bicarbonate,  
potassium oxalate, potassium nitrate and strontium salts;

anti-calculus agents, e.g. alkali-metal pyrophosphates,  
30 hypophosphite-containing polymers, organic phosphonates and  
phosphocitrates etc.;



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biomolecules, e.g. bacteriocins, antibodies, enzymes, etc.;

flavours, e.g. peppermint and spearmint oils;

5

proteinaceous materials such as collagen;

preservatives;

10 opacifying agents;

colouring agents;

pH-adjusting agents;

15

sweetening agents;

pharmaceutically acceptable carriers, e.g. starch, sucrose,  
water or water/alcohol systems etc.;

20

surfactants, such as anionic, nonionic, cationic and  
zwitterionic or amphoteric surfactants;

particulate abrasive materials such as silicas, aluminas,

25

calcium carbonates, dicalciumphosphates, calcium  
pyrophosphates, hydroxyapatites, trimetaphosphates,  
insoluble hexametaphosphates and so on, including  
agglomerated particulate abrasive materials, usually in  
amounts between 3 and 60% by weight of the oral care  
30 composition.

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humectants such as glycerol, sorbitol, propyleneglycol, xylitol, lactitol etc.;

binders and thickeners such as sodium carboxymethyl-  
5 cellulose, xanthan gum, gum arabic etc. as well as synthetic polymers such as polyacrylates and carboxyvinyl polymers such as Carbopol®;

polymeric compounds which can enhance the delivery of active  
10 ingredients such as antimicrobial agents can also be included. Examples of such polymers are copolymers of polyvinylmethylether with maleic anhydride and other similar delivery enhancing polymers, e.g. those described in DE-A-3,942,643 (Colgate);

15 buffers and salts to buffer the pH and ionic strength of the oral care composition; and

other optional ingredients that may be included are e.g.  
20 bleaching agents such as peroxy compounds e.g. potassium peroxydiphosphate, effervescing systems such as sodium bicarbonate/citric acid systems, colour change systems, and so on.

25 Liposomes may also be used to improve delivery or stability of active ingredients.

The oral compositions may be in any form common in the art, e.g. toothpaste, gel, mousse, aerosol, gum, lozenge, powder,  
30 cream, etc. and may also be formulated into systems for use in dual-compartment type dispensers.

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EXAMPLE 1

The bleaching agents were evaluated as follows:

- (1) Synthetic hydroxyapatite discs were polished and placed in sterile saliva at 37°C overnight to form a pellicle.
- (2) Discs were stained with tea solution for seven days at 37°C.
- (3) Stained discs were immersed in bleaching solutions for desired time.
- (4) The change in colour of the discs was measured using a Minolta chromameter CR-300 in L\*a\*b\* mode. Using L\* (initial), L\* (soiled), and L\* (cleaned), the percentage of stain removed was calculated. A negative value indicates a darkening and a positive value indicates a bleaching (whitening) effect.

All formulations used were made up in 0.5 M NaHCO<sub>3</sub>. A negative value indicates a darkening and

Table 1 below shows the use of picolinic acid alone and together with either 0.1 % H<sub>2</sub>O<sub>2</sub> or PAP.

|   | % Stain Removed |
|---|-----------------|
|   | 10 mins         |
| 0.1% H <sub>2</sub> O <sub>2</sub>          | 2               |
| 0.5 0.1 % H <sub>2</sub> O <sub>2</sub> /5% | 13              |

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|                          |    |
|--------------------------|----|
| picolinic acid           |    |
| 1% PAP                   | 27 |
| 1% PAP/5% picolinic acid | 46 |
| 5% picolinic acid        | 8  |

The results show in the table, show that the addition of a picolinic acid to the peracid provides a more effective  
5 bleaching composition over picolinic acid or the peracid alone.

**EXAMPLE 2**

- 10 The following oral composition comprises two formulations: a PAP-gel formulation and a pH-adjusting formulation.

**PAP-gel formulation**

15

| Ingredient       | Amount (% w/w) |
|------------------|----------------|
| PAP              | 0.6            |
| Gantrez          | 3.0            |
| Xanthan          | 0.2            |
| Silica           | 9.0            |
| Sodium hydroxide | 0.5            |
| Water            | to 100         |

PAP is commercially available as Eureco HC-17 (ex. Ausimont).

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This formulation has a pH of 4.

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pH-adjusting formulation

| Ingredient             | Amount % (w/w) |
|------------------------|----------------|
| Gantrez                | 3.0            |
| Xanthan                | 0.2            |
| Silica                 | 6.0            |
| picolinic acid         | 5.0            |
| Saccharin              | 0.25           |
| pH agent (TSP or NaOH) | to pH 11       |
| KNO <sub>3</sub>       | 5.0            |
| NaF/SMFP               | to 1500ppm     |
| Colour                 | as appropriate |
| Water                  | to 100         |

- 5 When both the PAP-formulation and the pH-adjusting formulation are mixed together the pH of the resulting composition is between about 6.5 and 8.5.

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**CLAIMS**

1. An oral composition comprising picolinic acid and a peroxy species or equivalent source thereof,  
5 characterised in that the molar ratio of picolinic acid to peroxy species or equivalent source thereof is from 1:30 to 100:1.
2. An oral composition as in claim 1, wherein the peroxy  
10 species or equivalent source thereof is a peracid or a compound that produces hydrogen peroxide on dissolution in water.
3. An oral composition according to any of preceding  
15 claims and comprising an additional transition metal chelator.
4. A deodorant product according to any preceding claim,  
20 wherein the composition comprises a transition metal chelator having a binding coefficient for iron (III) of greater than  $10^{15}$ .
5. An oral composition according to any preceding claim,  
25 wherein the composition comprises diethylenetriaminepentaacetic acid as a transition metal chelator.
6. An oral composition according to any of the preceding  
30 claims, wherein the molar ratio of picolinic acid to peroxy species or equivalent source thereof is from 1:10 to 20:1.

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7. An oral composition according to any of the preceding claims, wherein the picolinic acid is present at a level from 0.01% to 10% by weight of the composition of which it is a part.
8. An oral composition according to any preceding claim, wherein the picolinic acid is present at a level from 0.15% to 2.5% by weight of the composition of which it is a part.
9. An oral composition according to any of the preceding claims, wherein the peroxy species or equivalent source thereof is present at a level from 0.0001% to 1.5% by weight of the composition of which it is a part.
10. An oral composition according to any preceding claim, wherein the peroxy species or equivalent source thereof is present at a level from 0.003% to 0.5% by weight of the composition of which it is a part.
11. An oral composition according to any of the preceding claims and comprising a cosmetically acceptable carrier material.
12. Use of an oral composition according to any preceding claim for a treatment selected from the group consisting of tooth whitening, anti-plaque, anti-malodour, anti-caries and anti-tartar.



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13. Use of a composition according to any preceding claim  
in the manufacture of a medicament for a treatment  
selected from the group consisting of tooth whitening,  
5 anti-plaque, anti-malodour, anti-caries and anti-  
tartar.

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 01/14496

**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC 7 A61K7/16 A61K7/20 A61K7/22

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category * | Citation of document, with indication, where appropriate, of the relevant passages              | Relevant to claim No. |
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|            | -/--  |                       |

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Form PCT/ISA/210 (continuation of second sheet) (July 1992)

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 01/14496

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